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치의학박사학위논문

Effect of various intermediate ceramic  
layers on the interfacial stability between  
zirconia core and veneering ceramics

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윤 형 인

-ABSTRACT-

# Effect of various intermediate ceramic layers on the interfacial stability between zirconia core and veneering ceramics

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**Objectives:** The purposes of this study were to evaluate the effects of intermediate ceramics on the adhesion between the zirconia core and veneer ceramics.

**Materials and methods:** The polished surfaces of fully sintered Y-TZP blocks received three different treatments: 1) connector (C), 2) liner (L), or 3) wash layer (W). All the treated zirconia blocks were veneered with either a) fluorapatite glass-ceramic (E) or b) feldspathic porcelain (V) and divided into four groups (CE, CV, LE and WV). For the

control group, the testing surfaces of metal blocks were veneered with feldspathic porcelain (VM). All veneered blocks were sectioned into bars (3 mm × 3 mm × 7 mm), perpendicularly to the interface. A half of the samples in each group ( $n = 21$ ) were exposed to thermocycling for 20,000 cycles. The temperature was altered between 5°C and 55°C with an immersion time of 30 seconds and a transfer time of 2 seconds. The other half of the specimens were stored at room temperature under dry conditions. All specimens were subjected to the shear test, and the failed surfaces were microscopically examined. The elemental distribution at the zirconia core/veneer interface was analyzed. The bond strength data of the groups were compared with one-way analysis of variance (ANOVA) and the post hoc analyses were conducted using a Tukey test ( $\alpha=0.05$ ).

**Results:** The specimens in Group CE and CV exhibited significantly greater mean bond strength values than those in Group LE and WV, respectively ( $P<0.05$ ). However, the mean bond strengths significantly decreased in the connector groups (CE and CV) after thermocycling ( $P<0.05$ ). The elemental analysis suggested diffusion of ceramic substances into the zirconia surface.

**Conclusions:** A glass-ceramic based connector is significantly more favorable to core/veneer adhesion than the other intermediate ceramics evaluated in the study. However, thermocycling affected the bond strength at the core/veneer interface differently according to the intermediate ceramics.

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**Keywords:** Zirconia ceramic, Veneer ceramic, Intermediate ceramic, Thermocycling, Interface

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# Effect of various intermediate ceramic layers on the interfacial stability between zirconia core and veneering ceramics

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KOREAN ABSTRACT

# **I. INTRODUCTION**

Metal–ceramic restorations are widely used in prosthetic treatments because of their good mechanical properties and clinically acceptable qualities [1, 2]. However, since these restorations lack translucency, a layer of opaque porcelain is required to mask the unesthetic metal shade, and this necessitates the development of metal-free prostheses for desirable esthetic outcome [3]. With the development of high strength ceramics and the progress in CAD/CAM technology, yttrium oxide stabilized tetragonal zirconia polycrystal (Y-TZP) is being increasingly used as a core material for all-ceramic restorations in clinical practice and research. Previous studies have shown that zirconia exhibits several advantages such as esthetic appearance, biocompatibility, wear resistance, high flexural strength, and fracture toughness [3].

As with conventional metal–ceramic restorations, many of the zirconia-based all-ceramic systems consist of a substrate and veneering ceramic to yield tooth-like optical properties and optimal esthetic outcome [4]. Manufacturers recommend the use of feldspathic ceramics and nanofluorapatite glass ceramics as veneers on the surfaces of the Y-TZP frameworks [5]. In fact, the failure of zirconia-based prostheses is mainly attributed to mechanical problems such as the chipping and delamination of veneering ceramics [3]. As reported by a systematic review, zirconia-based restorations exhibit a five-year mean survival rate of 94.3% and a complication free rate of 76.4% for the same period [6]. Incidences of veneer chipping in zirconia-based restorations are found to range

from 21% to 32%, over 5 to 10 years [7-9]. In contrast, the fracture rate of veneer porcelain for metal-based ceramic restorations is reported to be 2.9%, which is significantly lower than that for zirconia-based prostheses [1, 6, 10, 11]. Researchers have suggested various reasons for the chipping and delamination of veneer material, such as 1) differences in coefficients of thermal expansion (CTE) between the framework and veneering porcelain, 2) areas of porosities, 3) mechanical defects in the porcelain or zirconia, 4) poor wetting by the veneering material on the core, 5) improper framework support, and 6) low thermal conductivity of zirconia [3, 11].

Along with the thermal compatibilities between the core and veneering materials, additional adhesive methods by mechanical or chemical modifications on zirconia surfaces have been proposed to ensure stable bonding at the core/veneer interface [12-14]. Treatment of the surfaces with airborne-particle abrasion may increase surface irregularities by providing an enlarged area for micromechanical interlocking with veneering material [15, 16]. The application of a liner can also improve the contact between the materials by building up an intermediate layer at the interface [17]. Recently, a glass-ceramic based connector (Hotbond Zirconnect) has been used for bonding between zirconia core and various ceramic systems. To the best of our knowledge, there are few comparative studies on the bond strengths at the interface with these intermediate ceramics under thermocycled conditions, which are more similar to clinical situations than dry experimental conditions.

The objective of this study was to evaluate the effects of three different intermediate



layers including connector, liner, and wash layer of veneering ceramics, on the bonded interface between the zirconia core and two commercial veneer ceramic systems. The shear bond strengths of the core/veneer complexes were evaluated after dry storage and after thermocycling. The metal–ceramic bilayers served as references. The null hypotheses were that, regardless of applied intermediate ceramics, the bond strength values of veneer ceramics to Y-TZP substrate could not be different from that to metal substrate, and the bond strengths of all the complexes would not be affected by thermocycling.

## **II. MATERIALS AND METHODS**

The chemical compositions and batch numbers of the materials used in this study are presented in Table 1.

### **2.1 Preparation of core specimen**

Pre-sintered, 3 mol% yttria-stabilized zirconia blocks (ZirBlank-PS, Acucera, Pocheon, South Korea) were sintered at 1500°C for 2 hours in a furnace (Austromat Basic, Dekema) and prepared with 24 equal sized cuboidal blocks (15 mm × 15 mm × 4 mm). A square face of each block, intended for bonding, was polished to obtain a standardized surface and cleaned in an ultrasonic bath with ethanol for 5 minutes. In addition, 42 metal blocks (5 mm × 5 mm × 4 mm) were cast using a nickel-chromium alloy (Bellabond plus) according to the manufacturer's instructions. One square surface of each metal block was conditioned with airborne-particle abrasion (Basic master, Renfert) using 125-μm aluminum oxide (Cobra, Renfert, Hilzingen, Germany) at a pressure of 0.4 MPa. The surfaces were steam-cleaned and air-dried. Oxidation treatment was subsequently performed in a furnace (Austromat D4, Dekema, Freilassing, Germany) according to the manufacturer's instructions.

### **2.2 Application of intermediate ceramic layer**

The polished surfaces of fully sintered Y-TZP blocks received three different treatments

as intermediate ceramic layers: 1) connector (C), 2) liner (L), or 3) wash layer (W) (Fig. 1). All the treated zirconia cores were veneered with either a) fluorapatite glass-ceramic (E) or b) feldspathic porcelain (V) and divided into four experimental groups (CE, CV, LE and WV). For the control group, the testing surfaces of metal blocks were veneered with feldspathic porcelain (VM). For Group CE, each of the polished zirconia surfaces received airborne-particle abrasion (Basic master, Renfert) with 125- $\mu$ m aluminum oxide (Cobra, Renfert) at a pressure of 0.2 MPa from a distance of 10 mm. A powder of the connector (Hotbond Zirconnect) was mixed with a liquid (Hotbond carrier liquid, DCM, Rostock, Germany) to achieve a watery consistency, and this mixture was sprayed on the roughened surface of each block. The firing process was carried out according to the manufacturer's instructions to obtain a glassy surface (Austromat D4, Dekema, Freilassing, Germany) (Table 2). After the firing, the coated surface received additional surface abrasion with 125- $\mu$ m aluminum oxide (Cobra, Renfert, Hilzingen, Germany) at a pressure of 0.1 MPa as recommended by the manufacturer. Identically equal to Group CE, the application of the connector was repeated on the prepared surface of each zirconia block of Group CV. For Group LE, each of the polished zirconia surfaces was layered evenly with a liner (IPS e.max Ceram Zirliner). The specimens were then fired in a furnace according to the instructions of the manufacturers. For Group WV, the polished surfaces were coated with a thin aqueous mixture of feldspathic ceramic powder (VM 9) and a liquid (VM modelling liquid, Vita Zahnfabrik, Bad Säckingen, Germany), and this 'wash layer' was fired according to the manufacturer's guidelines (Table 2).

### **2.3 Ceramic veneering**

For Groups CE and LE, the prepared surface of each block was then veneered with a fluorapatite glass ceramic (IPS e.max Ceram). For Groups CV and WV, the testing surfaces of the specimens were veneered with a feldspathic ceramic (VM 9). For Group VM, the oxidized surface of metal block was covered with a feldspathic ceramic (VM Opaque and VM 13). The build-up process was repeated to achieve a final veneering thickness of 3 mm. All specimens were fired in a furnace (Austromat D4, Dekema, Freilassing, Germany), according to the respective instructions of the manufacturers.

### **2.4 Preparation of bilayer test specimen and thermocycling**

All the veneered blocks were sectioned with a water-cooled diamond saw, perpendicular to the interface and trimmed into bars (3 mm × 3 mm × 7 mm). There were no premature failures in this process. A total of 168 zirconia-based specimens and 42 metal-based specimens were prepared. A half of the specimens in each group (five groups,  $n = 21$  per group) were subjected to thermocycling for 20,000 cycles. The water temperature was varied between 5°C and 55°C with an immersion time of 30 seconds and a transfer time between baths of 2 seconds. The other half of the specimens were stored at room temperature (20°C) under dry conditions. For both dry and thermocycled conditions, one representative sample of each test group was reserved for the elemental composition analysis of the core/veneer interface.

## **2.5 Shear bond strength test**

All specimens were placed in customized stainless steel molds and mounted on a test jig, before being loaded in a universal testing machine (Model 3345; Instron, Canton, MA, USA). A load was applied perpendicular to the long axis of the specimen, directly at the interface, using a chisel-shaped piston at a crosshead speed of 0.5 mm/min until failure occurs. The shear bond strength (MPa) was calculated by dividing the load at fracture (N) by the area of the bonded surface (mm<sup>2</sup>).

## **2.6 Microscopic examination of the fracture surface and elemental analysis of the interface**

After the bond strength test, three representative fracture specimens were cleaned ultrasonically and coated with platinum by ion sputtering for 30 seconds. The fractured surfaces were examined by field emission scanning electron microscopy (FE-SEM, S4700, Hitachi, Tokyo, Japan) with a 15 kV accelerating voltage. The modes of failure were classified as follows: cohesive in the veneering ceramic, adhesive at core-veneer interface, and mixed. Each interfacial surface was analyzed by energy dispersive X-ray spectroscopy (EDS), and the weight percentage of every traced element was measured (EX-250, Horiba, Kyoto, Japan). In addition, one representative veneered specimen of each test group was analyzed for the elemental distribution around the bonded interface. One axial surface of each specimen was polished and ultrasonically cleaned to remove external irregularities. A line profile analysis was performed over a distance of 13  $\mu$ m

from the side of the interface on the specimens. The lateral resolution was estimated at 1  $\mu\text{m}$ .

## **2.7 Statistical analyses**

The shear bond strength data of the groups were compared with one-way analysis of variance (ANOVA) and the post hoc Tukey multiple comparisons method. The mean shear bond strength values for between specimens subjected to dry storage and thermal cycling for all experimental and control groups were compared using the independent t-test. The statistical analyses were performed with the Statistical Package for the Social Sciences (SPSS v.21.0, Inc., Chicago, IL, USA). The level of statistical significance ( $\alpha$ ) was set as 0.05.

### III. RESULTS

The results of the shear bond strength tests are shown in Table 3. None of the samples showed early fractures of the veneering ceramic during the aging process. Among the Y-TZP specimens subjected to dry storage, Group CV exhibited significantly higher mean bond strength compared to the metal–ceramic group (VM), whereas the Group LE exhibited significantly lower mean bond strength. For the groups with the same veneering ceramic, the connector significantly improved bond strength at the interface compared with other intermediate ceramics used for adhesion. Groups CE and CV exhibited significantly greater mean bond strength values compared to Groups LE and WV, respectively. After thermocycling, the mean bond strength value of Group CE was significantly lower than that of the metal–ceramic group. The mean values of shear bond strengths of the other zirconia groups did not significantly differ from that of the control group. The mean bond strength values of the groups having the connector (Groups CE and CV) decreased significantly after thermal aging, while those of the groups with other intermediate ceramics did not.

Every zirconia specimen exhibited mixed pattern of fracture, which included cohesive fracture within the veneering material and concurrent adhesive fracture at the interface, irrespective of thermocycling (Fig. 2a–2h). The fracture originated near the loaded area and propagated into the veneering ceramics, close to the bonding interface. Thin layers of glass ceramic materials were partially attached on the debonded zirconia surfaces. On the

contrary, the metal–ceramic specimens predominantly showed cohesive fractures within the veneering porcelain (Fig. 2i and 2j). The EDS spectrum at the fractured interface of Y-TZP specimen detected the existence of sodium, potassium, aluminum, and silica (Fig. 3).

For detailed elemental distribution, the line scan graph of all examined zirconia specimens showed a decrease in zirconium in the core to the veneering porcelain with a decrease in the silicon element in veneer ceramic to the core (Fig. 4). Along with the scanned line across the interface, the curve of zirconium concentration declined sharply into the veneer ceramic area, whereas the curve of silicon concentration showed a relatively gradual increase. For each zirconia/veneer ceramic complex, the area of elemental transition was observed, and its thickness was approximately 2–4  $\mu\text{m}$ .



## **IV. DISCUSSION**

This study evaluated the effect of three different intermediate ceramic layers on the adhesion between Y-TZP cores and veneering ceramics, and examined the susceptibilities of those bonded interfaces to thermocycling. The mean shear bond strength of the metal–ceramic specimens was significantly different from those of the experimental groups bonded with a connector or a liner. The application of the connector on zirconia surfaces tends to improve the interfacial bond strength with veneering porcelain to an extent similar to or greater than that of metal/ceramic bonding. Moreover, groups with the connector exhibited significantly stronger adhesion between the core and the veneer material compared to the groups with the liner or wash layer of veneering porcelain. The results of this study thus supported rejection of the primary null hypothesis. The secondary null hypothesis was also rejected since the thermocycling resulted in a significant decrease in the bond strength at the interface of the specimens layered with the connector. On the contrary, no significant influences were detected for the specimens with other intermediate ceramics regardless of exposure to thermocycling.

For the long-term success of Y-TZP all-ceramic restorations, three factors should be considered: adequate cooling rate, compatibility of the coefficient of thermal expansion (CTE) of the core and veneer ceramics, and strong adhesion between the two materials [5]. Some studies reported that a strong bond at the interface of the core and veneer material depends on the adequate wetting behavior of the solid by the liquid phase as well

as their thermal compatibility [15, 18]. When the CTE mismatch was in the range from  $0.75$  to  $1.7 \times 10^{-6} \text{ K}^{-1}$ , its influence on the core/veneer bond strength was not significant [5]. In this study, the veneering porcelain, the liner, and the connector had compatible CTE values with that of the Y-TZP core. The intermediate ceramic layers such as liner or connector may increase the wettability of the core surface as well as compensate for the discrepancy in the thermal expansion behavior between the veneer and the substrate [17, 19, 20]. The wetting property is determined by the composition of the adhesive material, the morphology of the zirconia surface, and the surface energy of the core [21]. In this study, the application of the connector at the interface between the veneer ceramic and the zirconia surface proved to be more effective than the other treatments. The connector may have better wetting behavior and higher elastic property compared to the other intermediate ceramics. Airborne-particle abrasion on the zirconia surface before and after application of the connector material may also have contributed to the strong interfacial adhesion. The irregularities in the core surface and the increased contact area with the veneering ceramic improve the mechanical retention and the interfacial bond strength values, thus reducing the chances of delamination [14, 15, 22].

Before the application of connector, the zirconia surface must be subjected to airborne abrasion with  $125\text{-}\mu\text{m}$  alumina particles at a pressure of  $0.2 \text{ MPa}$  according to the manufacturer's recommendation. This treatment, however, has been reported to cause structural transformation from tetragonal to monoclinic phase up to a penetration depth of  $27 \text{ }\mu\text{m}$  from the zirconia surface [23]. The difference in the CTEs of the monoclinic and

tetragonal phases can have a negative effect on the zirconia/veneer ceramic bond strength [24]. Some studies have reported that sandblasting surfaces of zirconia prior to veneering does not significantly improve the shear bond strength between the bilayer composites compared to non-treated or polished surfaces [5, 15]. Therefore, the bonded interface between zirconia core and veneer porcelain with the layer of connector should be examined in the future, considering the negative effect of phase transition on the surface.

Microscopic observation of the fractured Y-TZP surfaces revealed combined patterns of cohesive and adhesive failure, which correspond well with observations of previous research [5, 25, 26]. The failure mode of the metal–ceramic specimens was mainly cohesive, which is also consistent with previous results [5]. Delamination of veneer ceramic has been documented as adhesive failure at the bonded interface, although some studies have reported that the bond strength between the core and veneer material seems to be higher than the cohesive strength of the ceramic itself [13, 27]. Since zirconia has a high fracture toughness as a core material, the delamination of the veneering ceramic from undamaged Y-TZP substrate can result from deflected crack propagation at the core/veneer interface [5, 14]. Stable adhesion of the veneering ceramic to zirconia core, as well as favorable mechanical properties of veneer materials, is vital for ensuring resistance to interlaminar crack propagation [5].

Metal–ceramic composites have been reported to exhibit higher bond strengths than Y-TZP all-ceramic composites [5, 25, 26]. In this study, the mean strength values of the metal and zirconia group specimens are similar to or greater than the findings of the

previous studies and the standard bond strength of metal–ceramic composites [5, 12, 25, 26]. In fact, the bond strength of smaller specimens are higher than those of larger specimens because larger specimens more frequently exhibit surface defects and possess a higher risk of early bonding failure [12]. The use of the connector helped enhance the interfacial adhesion between the Y-TZP substrate and veneering ceramic, analogous to the degree of metal/ceramic bonding. However, the stress corrosion of ceramic materials and the resultant slow crack growth, facilitated by the complex aqueous situation of the oral cavity, should be considered in clinical dentistry [27]. Except for the Y-TZP core/veneer ceramic complexes bonded with the connector, the other zirconia and metal specimens were not significantly affected by thermal aging, which reaffirms the results of previous studies [5, 26, 28]. Cyclic loading of the bilayer specimens in hot and cold fluids can be a valuable testing method to examine the aging sensitivity to the bond strength at the core/veneer interface under exposure to thermal stress [5, 29]. Of all ceramics, silicate glasses are among the most susceptible to stress corrosion in aqueous environment [30]. Since the content of the glass matrix and the mechanical properties of intermediate ceramics are dissimilar, the connector material may be more susceptible to moisture-induced stress corrosion compared to the liner or wash layer of feldspathic porcelain.

The stable adhesion of the metal and veneering ceramics is determined by micromechanical interlocking, similarity in the coefficient of thermal expansion, and chemical bond resulting from optimal metal oxidation and mutual diffusion of ions at the interface [31]. On the contrary, the mechanism of zirconia-veneer ceramic bonding is not

fully identified. The possibility of mutual elemental diffusion of zirconium and silicon, the two main components of the core and veneer material, was previously suggested [14, 28]. It was reported that some elements in the veneer ceramic, including silica, sodium, aluminum and potassium, could diffuse into the surface layer of the zirconia substrate to a depth of 8–10  $\mu\text{m}$ , gradually decreasing in concentration with the increase in the distance from the interface [14]. Studies of microscopic analysis on the zirconia/feldspathic porcelain interface also revealed a mutual diffusion process with an estimated elemental transition area having a maximum thickness of 2  $\mu\text{m}$  [32]. It was also proposed that the mutual diffusion area tends to increase with the application of the liner [33]. In agreement with previous findings, the line profile analysis of EDS in the present study showed the transition of elements at the bonded interface. The width of transition zone around the interface was similar between the specimens with the liner and connector. The thermocycling did not significantly affect the interface morphology. However, a previous study has failed to prove the migration of elements at zirconia/porcelain interface, the concept of chemical bonding by mutual diffusion requires further research [20].

Many studies have attempted to evaluate the interfacial bonding quality between the Y-TZP substrate and veneering material through a common in vitro shear bond strength test [26, 28, 34, 35]. However, critical assessments of the conventional in vitro research methods have shown that conventional shear protocols fail to consider uniform distribution of shear stresses [36]. Traditional shear bond test methods can be compromised by the non-interfacial failure that initiates from surface tensile stresses

away from the bonded interface and consequential incorrect stress values [34]. In fact, a standardized testing method for bond strength of Y-TZP all-ceramic materials has not been developed [37]. Non-anatomical specimens do not represent the complex framework design of clinical use, and current findings only provide basic information. The size and shape of each specimen in the present study was standardized in order to minimize experimental variables and to control the direction of stress during the shear test along the planar interface of bilayer specimens. For a controlled comparison, a base metal alloy was selected as a reference group because of its similar characteristics with zirconia with respect to the elastic modulus and thermal expansion behaviors [38]. Although the experimental conditions were standardized, the analysis of the bonded interface through an in vitro test method is one of the limitations of this study.

## **V. CONCLUSIONS**

The application of a glass-ceramic based connector as an intermediate bonding layer between zirconia core and veneering ceramic is significantly more effective for improving interfacial adhesion than the other treatments tested in this study. However, thermal stress in a wet environment affected the bond strength at the core/veneer interface differently according to the intermediate ceramics.

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## TABLES AND FIGURES

Table 1. Chemical composition and physical property of the core and veneering materials according to the manufacturers' information

Material	Manufacturer	Batch No.	Chemical composition	CTE** ( $\times 10^{-6}/K$ )
ZirBlank-PS	Acucera, Pocheon, South Korea	108081802	ZrO <sub>2</sub> , Y <sub>2</sub> O <sub>3</sub> , HfO <sub>2</sub>	10.5
Bellabond plus	Bego, Bremen, Germany	72919	Ni, Cr, Mo	13.8
Hotbond Zirconnect	DCM, Rostock, Germany	130218	Unknown*	9.7
IPS e.max Ceram ZirLiner	Ivoclar Vivadent, Schaan, Liechtenstein	P11447	SiO <sub>2</sub> , Al <sub>2</sub> O <sub>3</sub> , Na <sub>2</sub> O, K <sub>2</sub> O, ZnO, CaO, P <sub>2</sub> O <sub>5</sub> , F, other oxides	9.8
IPS e.max Ceram	Ivoclar Vivadent, Schaan, Liechtenstein	L43142	SiO <sub>2</sub> , Al <sub>2</sub> O <sub>3</sub> , Na <sub>2</sub> O, K <sub>2</sub> O, CaO, P <sub>2</sub> O <sub>5</sub> , F, other oxides	9.5
VM9	Vita Zahnfabrik, Bad Säckingen, Germany	11160	SiO <sub>2</sub> , Al <sub>2</sub> O <sub>3</sub> , Na <sub>2</sub> O, K <sub>2</sub> O, B <sub>2</sub> O <sub>3</sub>	8.8-9.2
VM13 (Opaque)	Vita Zahnfabrik, Bad Säckingen, Germany	34940	SiO <sub>2</sub> , Al <sub>2</sub> O <sub>3</sub> , Na <sub>2</sub> O, K <sub>2</sub> O, CeO <sub>2</sub>	14.0
VM13	Vita Zahnfabrik, Bad Säckingen, Germany	36970	SiO <sub>2</sub> , Al <sub>2</sub> O <sub>3</sub> , Na <sub>2</sub> O, K <sub>2</sub> O	13.1-13.6

\*The manufacturer did not provide authors with specific information of the material.

\*\*CTE: Coefficient of thermal expansion

Table 2. Firing schedules of veneering ceramics on zirconia and metal core specimens according to the manufacturers' recommendations

Material	Pre-heating temperature (°C)	Pre-heating time (min)	Heating rate (°C/min)	Firing temperature (°C)	Holding time (min)
Hotbond Zirconnect	450	2	60	1000	1
IPS e.max Ceram ZirLiner	403	4	40	960	1
IPS e.max Ceram	403	4	40	750	1
VM9 (wash layer)	500	2	55	950	1
VM9	500	6	55	910	1
VM13 (Opaque)	500	2	75	920	1
VM13	500	6	55	880	1

Table 3. Mean shear bond strength values of the specimens after dry storage and thermocycling

Groups	Shear bond strength (MPa)	
	Dry storage	Thermocycling
<b>CE</b>	43.7 (7.0) A,a	33.0 (4.6) A,b
<b>LE</b>	32.3 (7.6) C,a	34.5 (8.1) A,a
<b>CV</b>	51.6 (7.0) B,a	42.2 (5.0) B,b
<b>WV</b>	41.3 (11.4) A,a	41.3 (8.3) B,a
<b>VM</b>	40.8 (8.1) A,a	39.9 (10.6) B,a

\* The numbers in the parentheses are standard deviations. Values followed by identical uppercase letters present no statistical differences between groups. Lowercase letters with the same subscript show no significant differences within groups. Group codes: CE, Y-TZP + connector + IPS e.max Ceram; LE, Y-TZP + liner + IPS e.max Ceram; CV, Y-TZP + connector + VM 9; WV, Y-TZP + wash layer (VM9) + VM 9; VM, Ni-Cr alloy + VM 13.

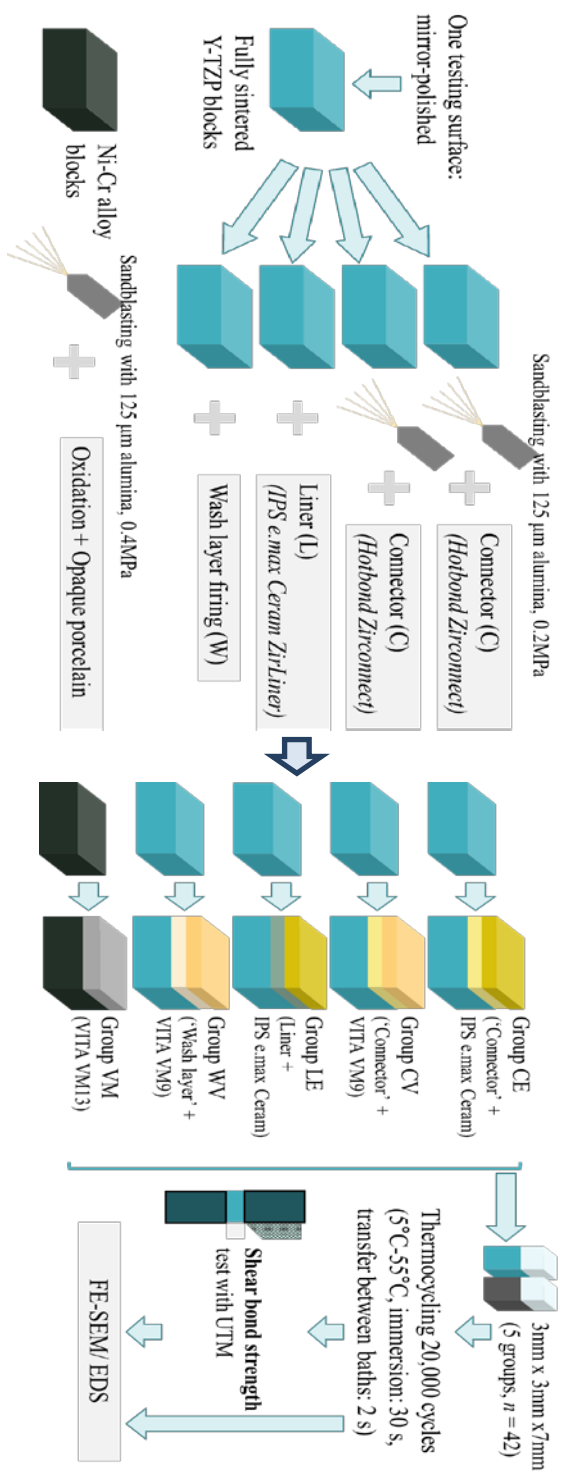
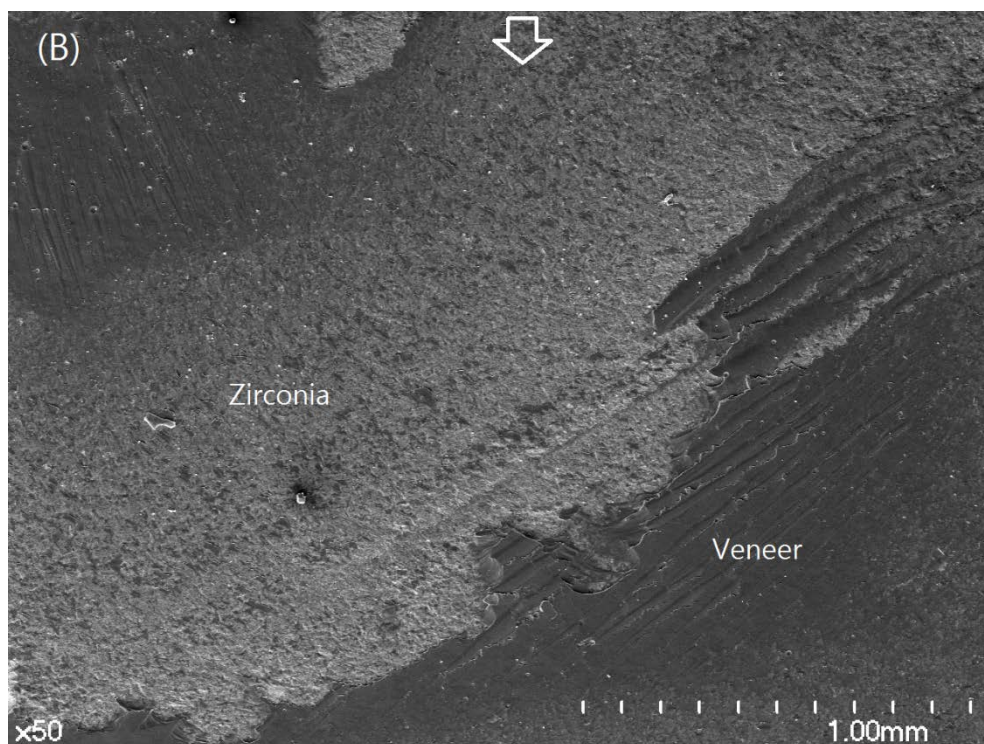
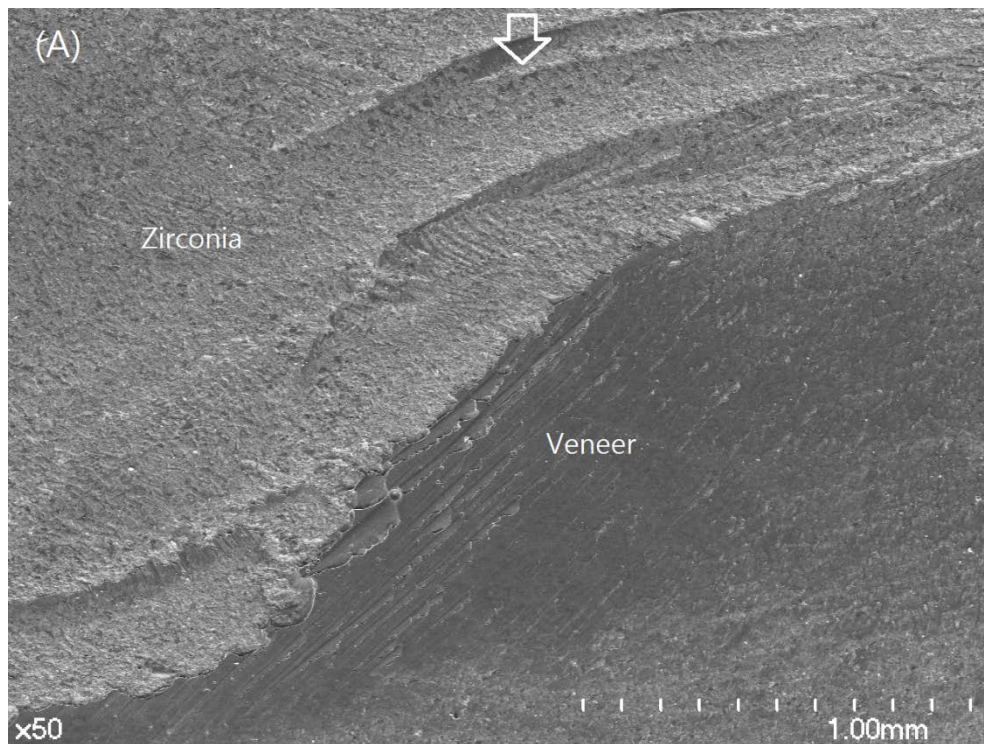
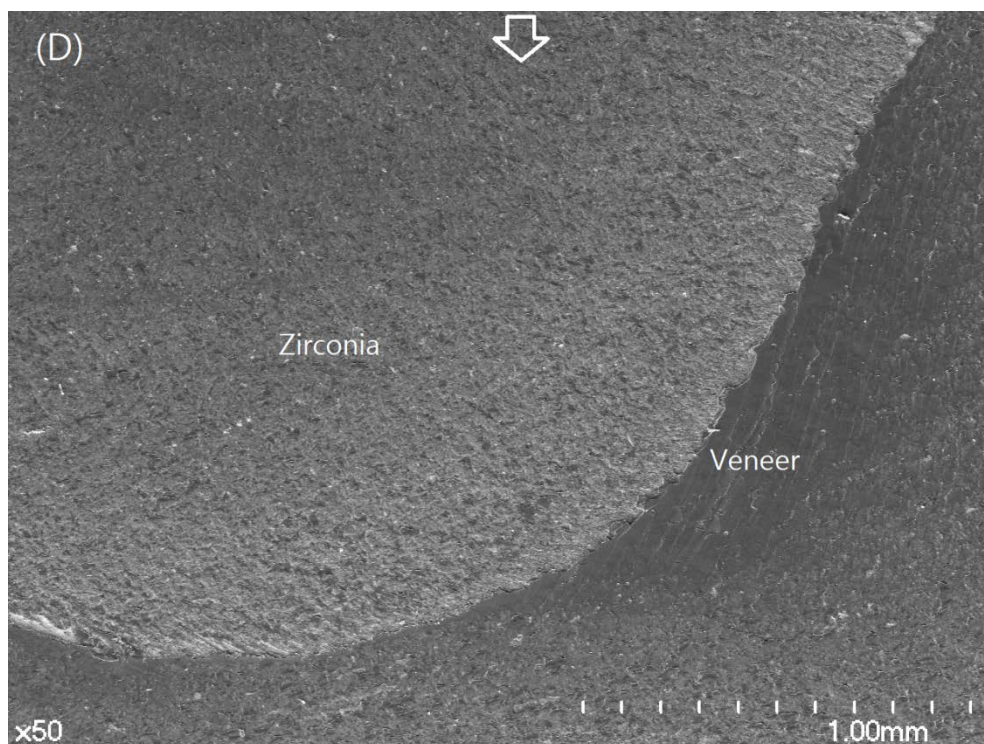
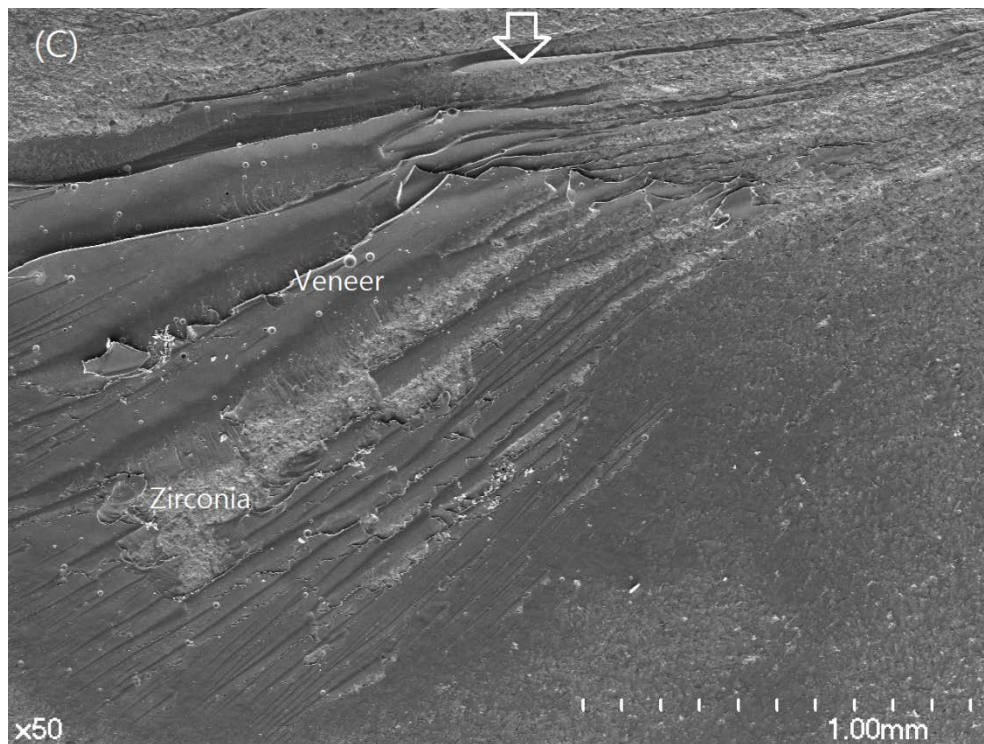


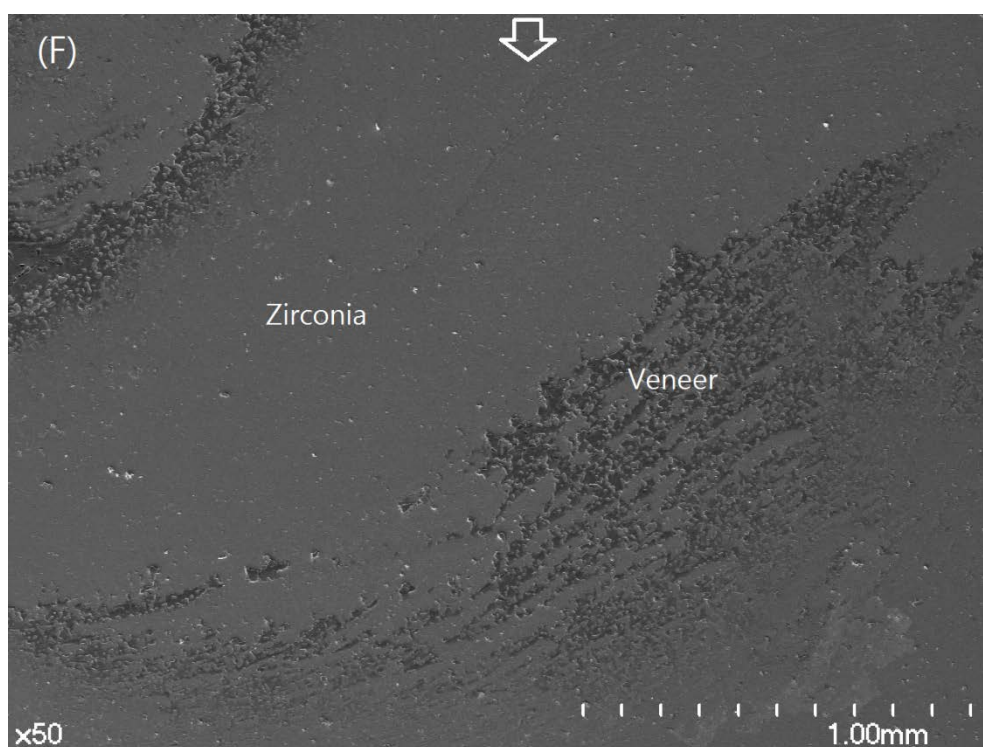
Fig. 1 - A schematic process-flow diagram of the specimens tested in this study.

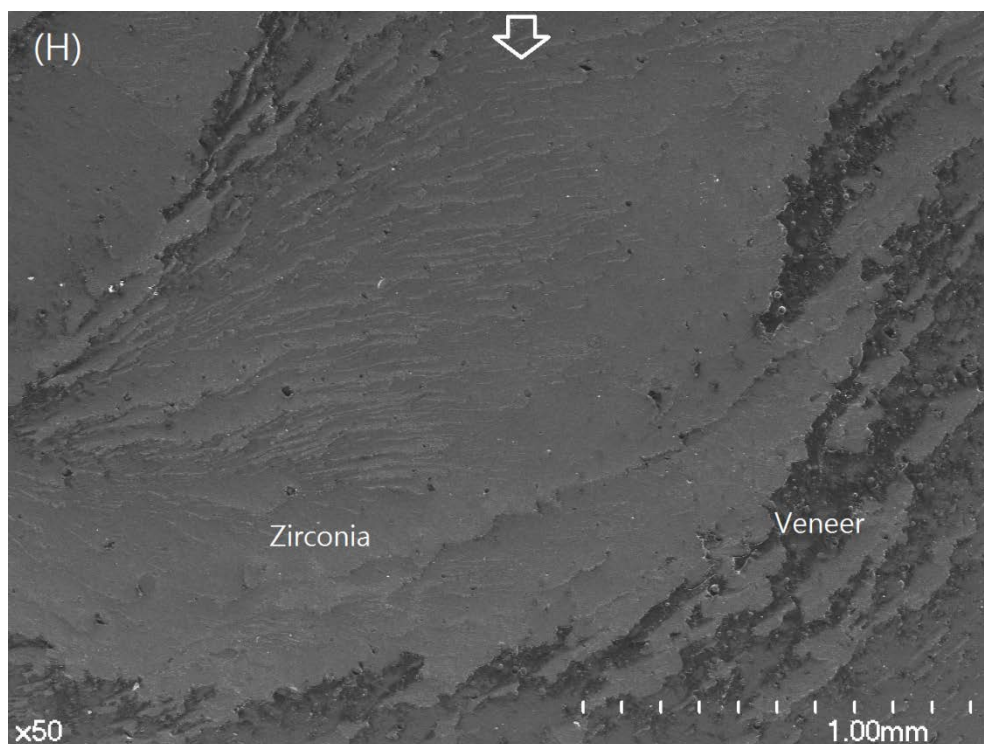
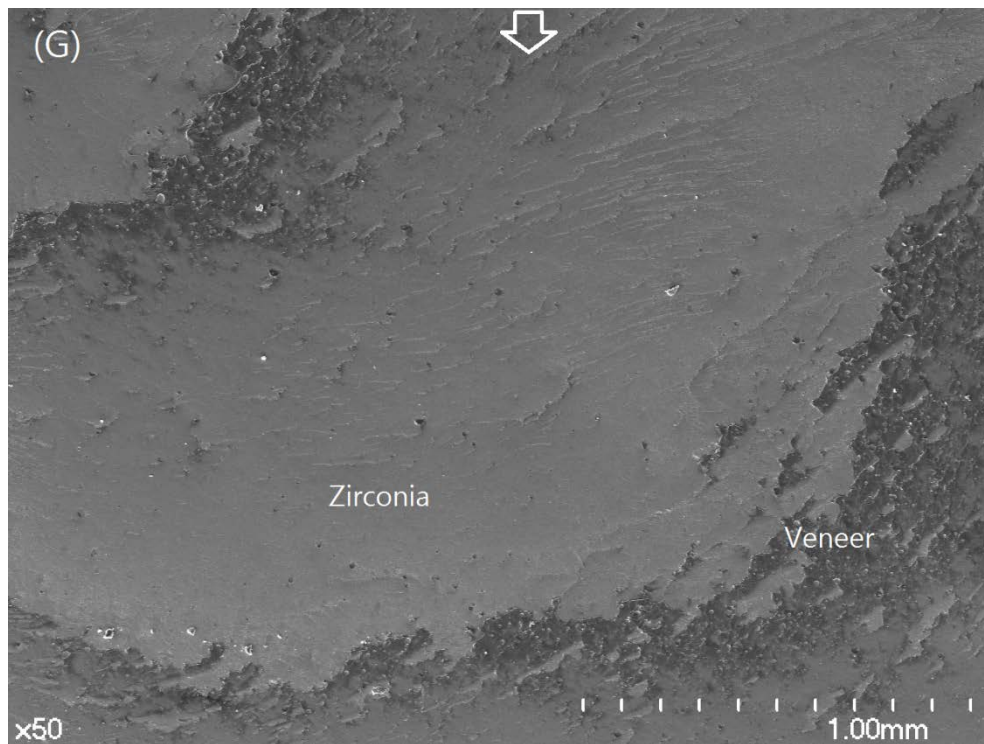














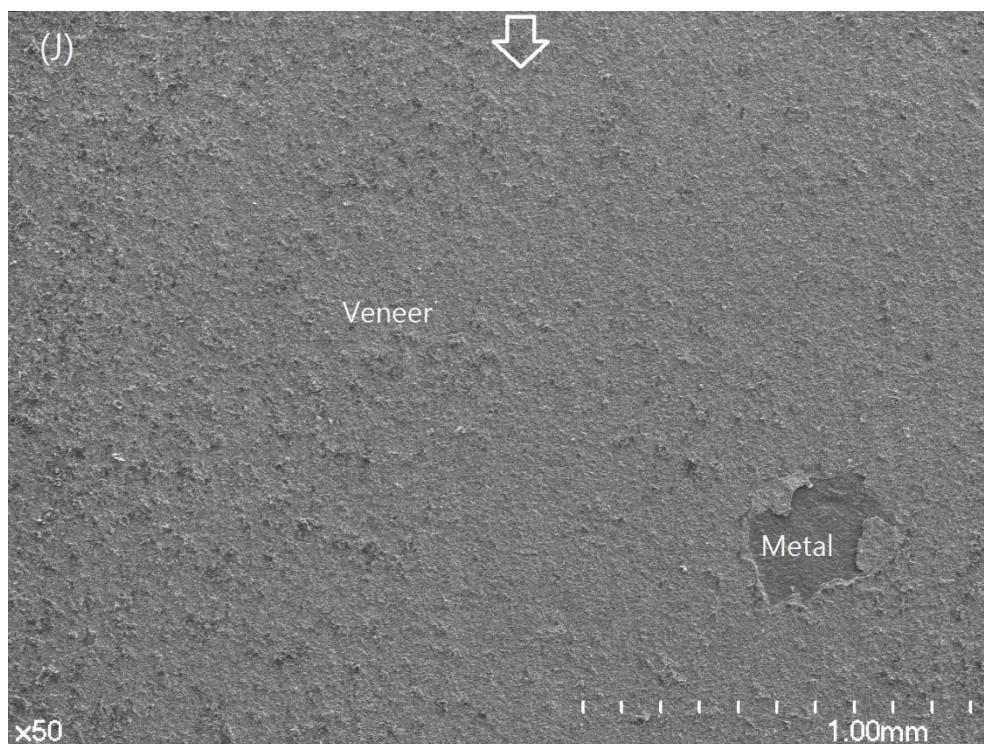
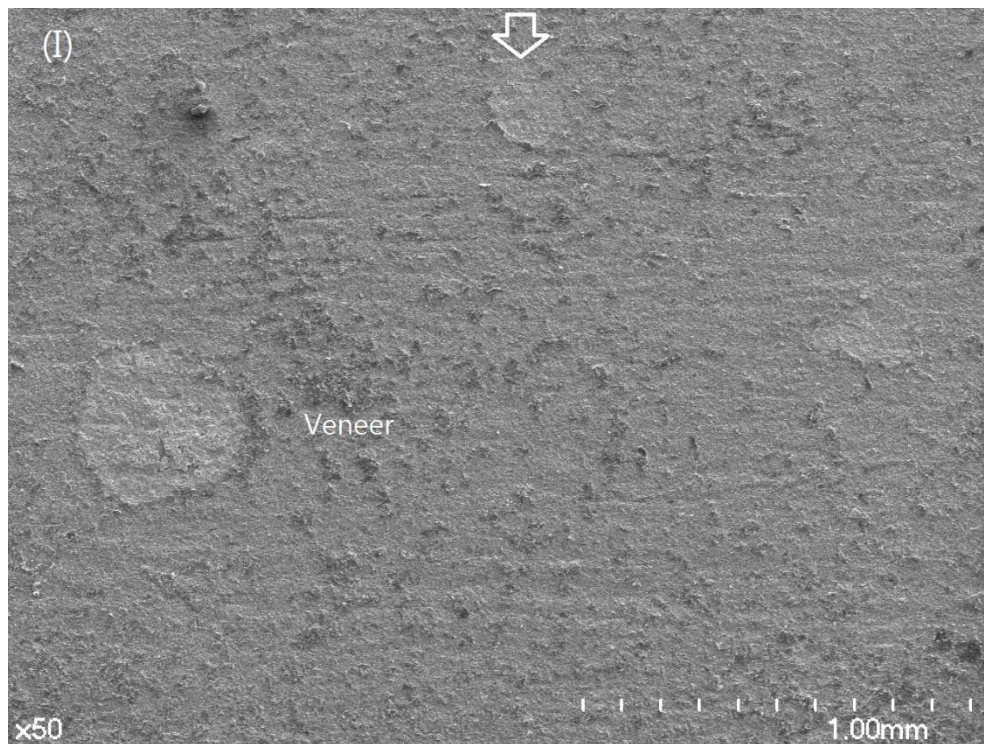


Fig. 2 - Representative SEM images of the fracture surfaces of Y-TZP and metal specimens. The arrow indicates the direction of shear force: A. Group CE, dry storage; B. Group CE, after thermocycling; C. Group CV, dry storage; D. Group CV, after thermocycling; E. Group LE, dry storage; F. Group LE, after thermocycling; G. Group WV, dry storage; H. Group WV, after thermocycling; I. group VM, dry storage; and J. group VM, after thermocycling.

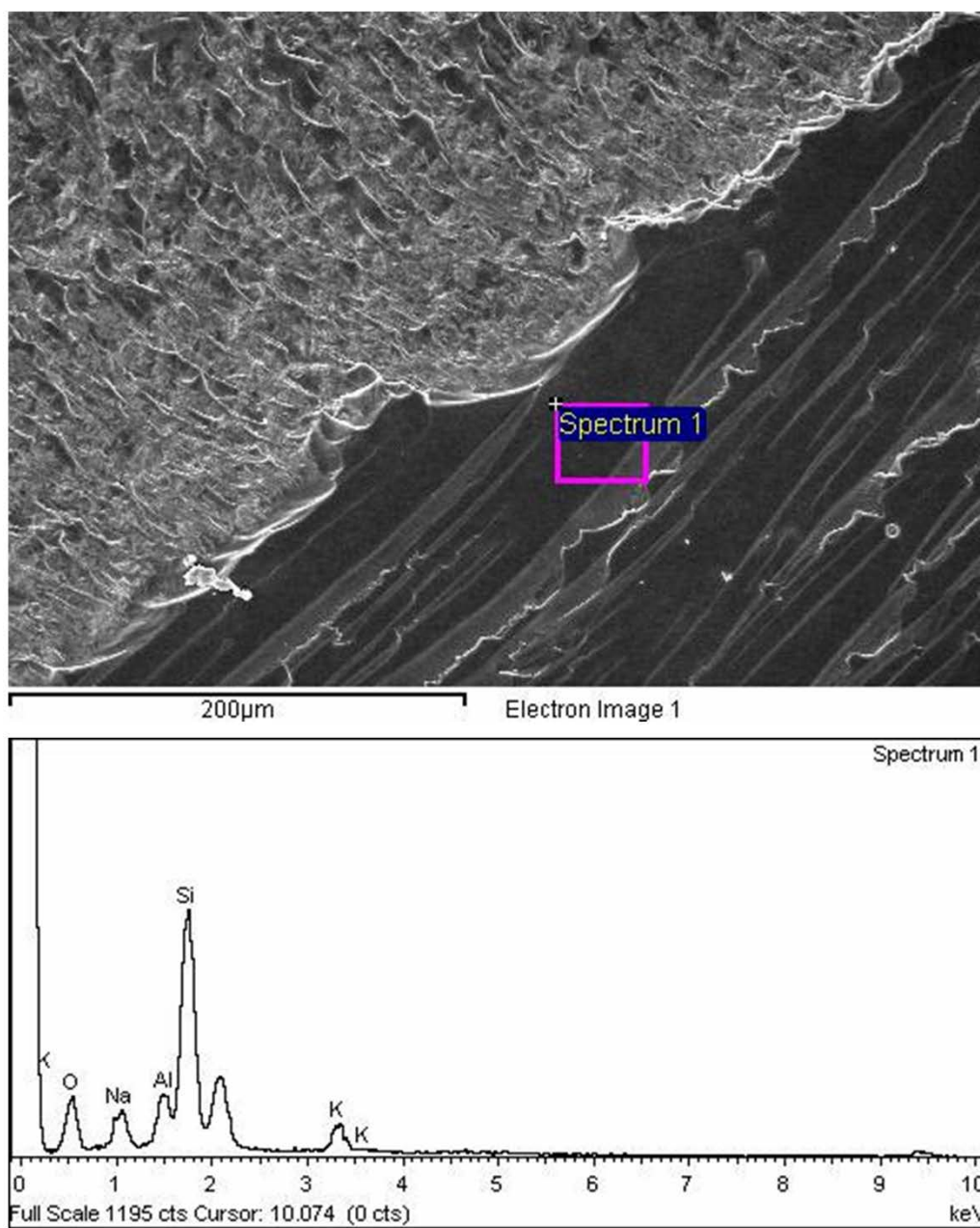


Fig. 3 - EDS spectrum at the fracture surface of Y-TZP core specimen (Group CV)



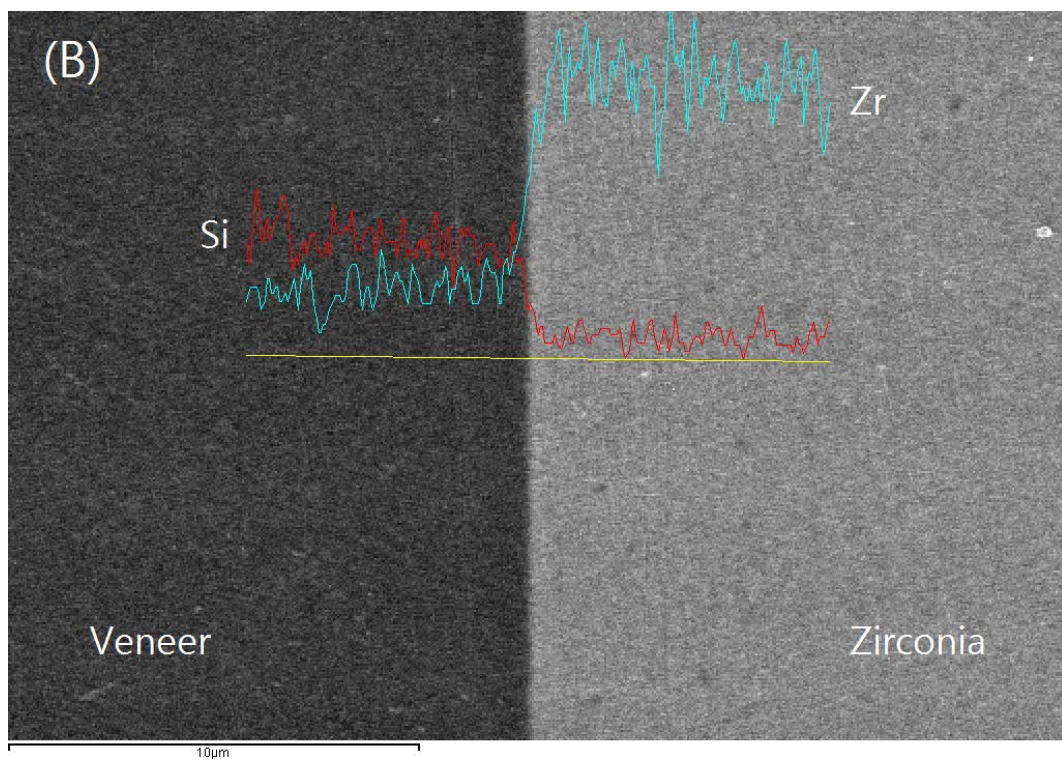
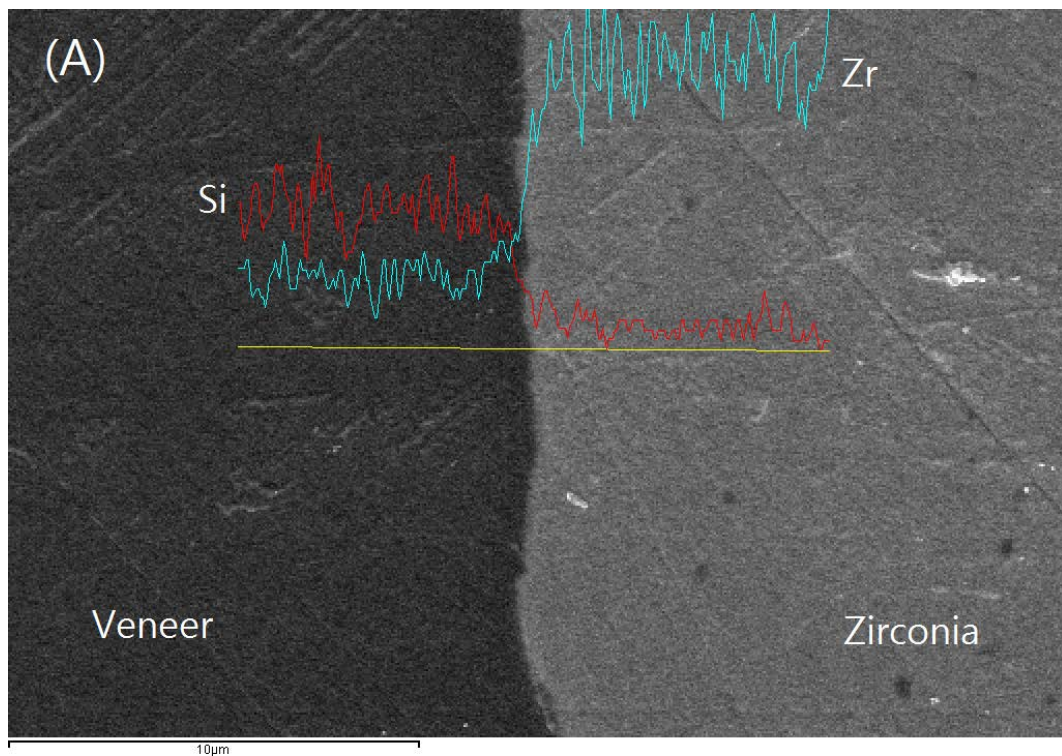




Fig. 4 - Elemental analysis across the Y-TZP core/veneer interface. Representative SEM images at the interface of the materials combinations are superimposed with the line scan graphs (blue line - zirconium, red line - silicon): A. Group CV, dry storage; B. Group WV, dry storage.

국문초록

다양한 결합용 도재 적용이 지르코니아 코어와 전장  
도재 사이의 결합 계면 안정성에 미치는 영향

서울대학교 대학원 치의과학과 치과보철학 전공

(지도 교수 한 중 석)

윤 형 인

**연구 목적:** 본 연구의 목적은 지르코니아 코어와 전장 도재 사이의 결합에 결합용 중간 도재가 미치는 영향을 평가하기 위한 것이다.

**재료 및 방법:** 완전 소성된 Y-TZP 블록의 한 면을 경면처리한 뒤 서로 다른 3가지 결합도재층(Hotbond Zirconnect (C), IPS e.max Ceram ZirLiner (L), VM9 wash layer (W))을 적용하였다. 이후 모든 지르코니아 시편들을 a) fluorapatite glass-ceramic (E) 또는 b) feldspathic porcelain

(V)으로 veneering 한 후 네 개의 실험군(CE, CV, LE, WV)으로 분류하였다. 대조군으로서 비귀금속 합금 코어를 feldspathic porcelain (VM)으로 veneering 하였다. 총 5개 군, 210개의 시편에서 각 군의 시편 중 절반 ( $n = 21$ )은 열순환처리를 하였고 나머지 절반은 상온에서 건조한 상태에 보관하였다. 이후 각 군당 두 개의 시편(건조 보관 1개, 열순환 처리 1개)을 제외한 나머지 모든 시편에 대하여 전단강도 실험을 진행하였고 파절된 표면은 현미경(SEM)으로 확인하였다. 강도실험에서 제외된 시편에 대해서는 지르코니아와 도재 간 결합계면에서 화학적 원소 분포를 분석하였다.

**결과:** 상온의 건조한 상태에서 CE 군과 CV 군의 시편들은 각각 LE 군과 WV 군의 시편들에 비하여 통계적으로 유의하게 높은 평균 전단강도 값을 나타내었다 ( $p < 0.05$ ). 그러나, 열순환 처리 이후 CE 군과 CV 군의 시편들의 평균 전단강도 값이 유의하게 감소하여, 처리 전후 강도 변화가 없는 다른 결합 도재군과 유사한 값을 나타내었다 ( $p < 0.05$ ). 파절면 분석 결과, 전장 도재의 세라믹 재료 성분이 지르코니아 코어 표면으로 침투하는 것으로 나타났다. EDS 분석 결과, 계면을 기준으로 Si 원소와 Zr 원소의 분포가 교차되는 혼합층이 약 2 ~ 4 마이크로 미터에 걸쳐 관찰되었다.

**결론:** 본 실험적 연구에서, 지르코니아와 전장 도재 간 결합을 위해 적

용된 중간 도재에 따라 계면에서 전단강도가 다르게 나타났으며, 특정 결합 도재가 제조사별 권장 결합재에 비해 더 우수한 효과를 보였다. 다만 구강 내처럼 수분이 있는 열역학적 환경에서 코어와 전장 도재 간 결합의 장기적 안정성은 결합 도재에 따라 다른 양상으로 나타나므로 지르코니아-도재 수복물을 위한 중간 도재 적용 시 이를 고려해야 한다.

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**주요어:** 지르코니아 세라믹, 전장 도재, 결합용 중간 도재, 열순환처리, 결합계면

**학번:** 2009-31122